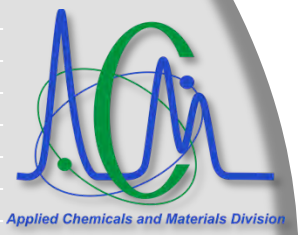


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# Materials Testing in Hydrogen Gas at NIST, Boulder*

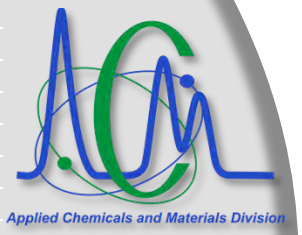
AJ Slifka, ES Drexler, RL Amaro,
DS Lauria, JR Fekete, K Eason

Sandia National Laboratories, Livermore, CA, April 9-10, 2013

# Outline

- Capabilities
- Design
- Transducers
- Test Procedures
- Safety
- Issues

# Objective

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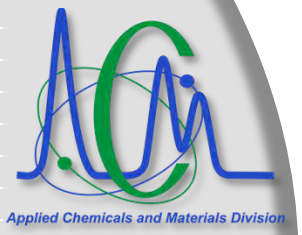
- Provide critical data and measurement methods that enable safe and economical transport of hydrogen fuel.
  - Our focus has been on fatigue crack growth testing
  - We may start some fracture testing this year
    - Have not done fracture testing in the past (in $H_2$)

# Design Approach

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- Followed the lead of Sandia
- New laboratory built for hydrogen purpose
- Custom test vessels (2)
- Servo-hydraulic load frames (2)
- Support Equipment for Remote Location
  - Used to have a chiller
  - Power electronics
  - Hydraulic power unit
  - Air compressor
  - Hydrogen sensors

# Laboratory

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- Class I, Division II, Group B
- 925 square feet, 12,000 cubic feet
- 350 cubic meters
- Maximum hydrogen gas volume = 6.4 cubic meters

# Test Capabilities

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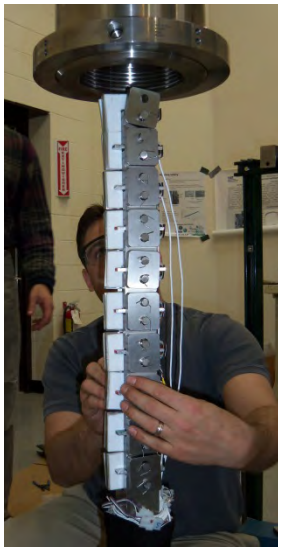
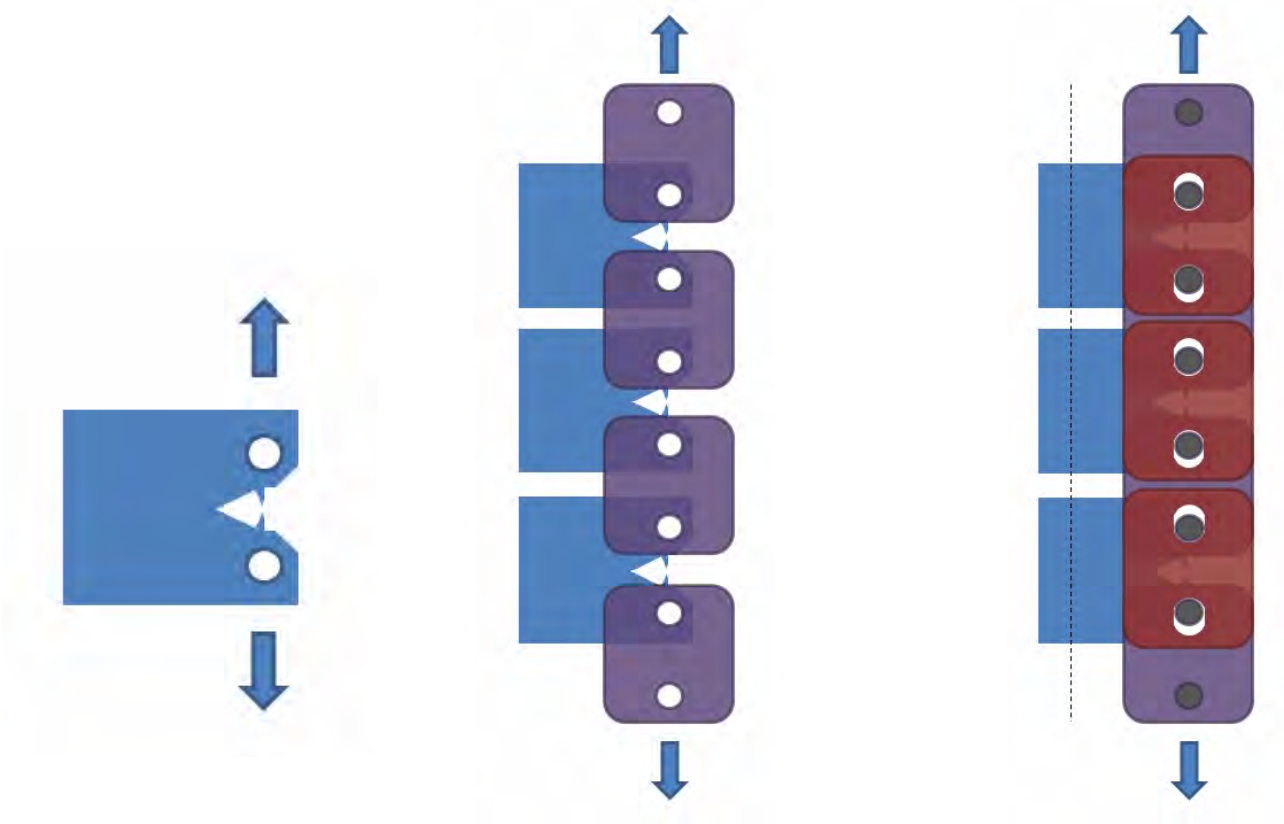
| | | | |
|---|---|---|---|
| Load Frame 1 | 100 kN, 22 kip | | |
| Load Frame 2 | 250 kN, 55 kip | | |
| Pressure Vessel 1 | 138 MPa, 20,000 psi | 0.55 $ft^3$, 1.5 liters | 4" x 7.5" |
| Pressure Vessel 2 | 34 MPa, 5000 psi | 0.341 $ft^3$, 9.6 liters | 5" x 32" |
| Room Temperature | | | |
| Test control | tensile, $10^{-6}$ | fatigue, 0.01 - 10 Hz | |



# Test Capabilities

- Multiple-specimen fatigue



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# Test Vessels

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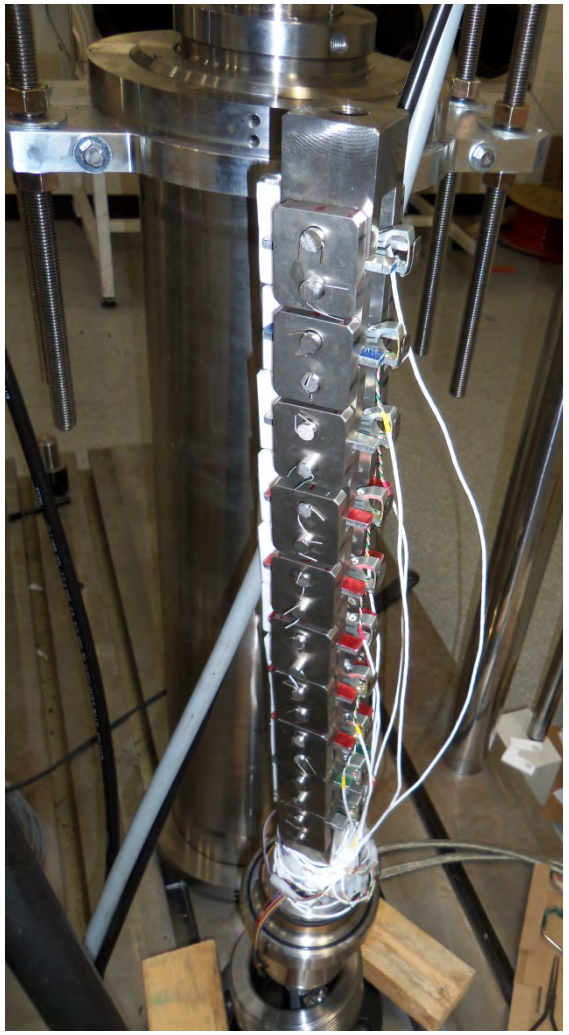
- Small vessel same as Sandia's
- Large vessel has inner length of 813 mm (32 in.) and inner diameter of 127 mm (5 in.), 316 SS, same end caps



# Transducers

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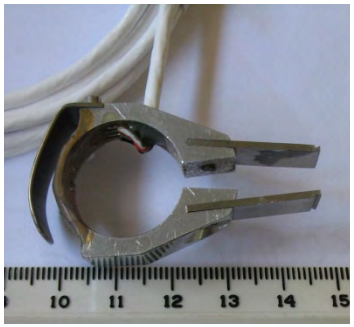
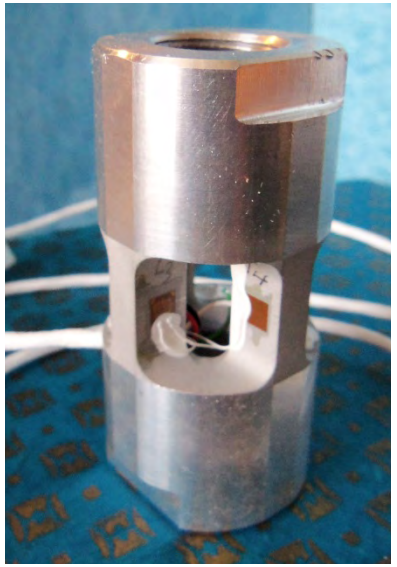
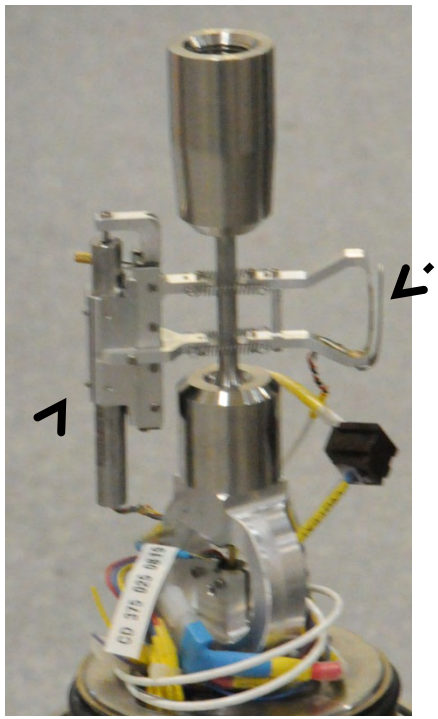


Strain-gauged clip gauge

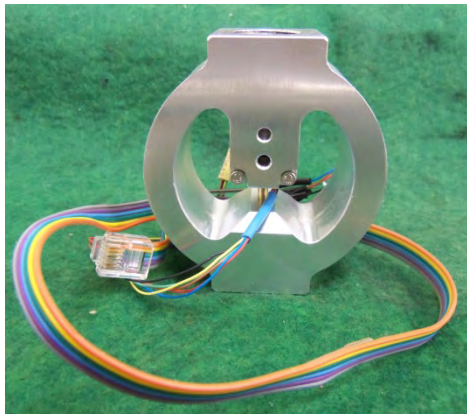


Strain-gauged 22 kN (5 kip) Load cell



Strain-gauged extensometer

Vented-type LVDT extensometer



44 kN (10 kip) proving ring load cell (vented LVDT)

# Hydrogen Compressors

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- 41 MPa (6000 psi)


- 150 MPa (22,000 psi)
- Both are diaphragm-type

# Gas Supply System

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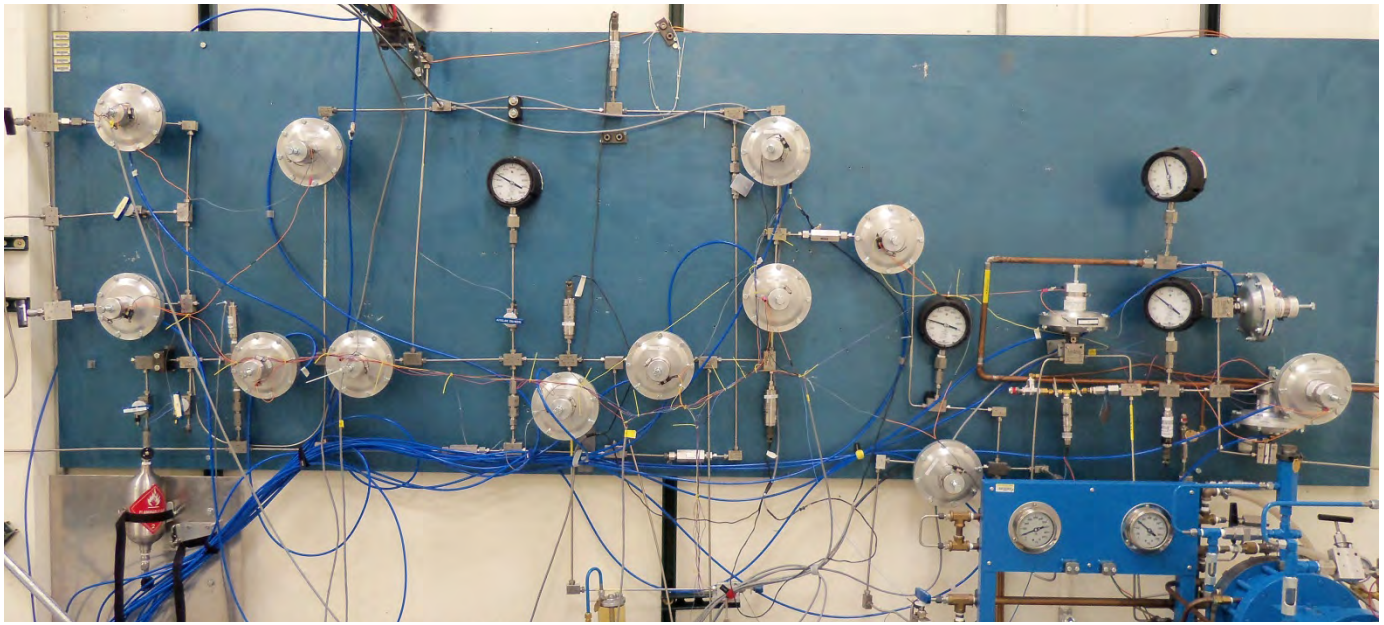
- LabVIEW* automated control
- Accumulator for each test chamber to lessen the load on the compressors
- Micro-needle valves used for de-pressurization at <69 kPa (10 psi) per minute
- Gas sampling of 1 liter, 6 MPa (900 psi) samples
- Supply interlocked to ventilation systems and hydrogen sensor alarm



* Commercial equipment, instruments, or materials are identified only in order to adequately specify certain procedures. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products identified are necessarily the best available for the purpose.

# Gas Supply System

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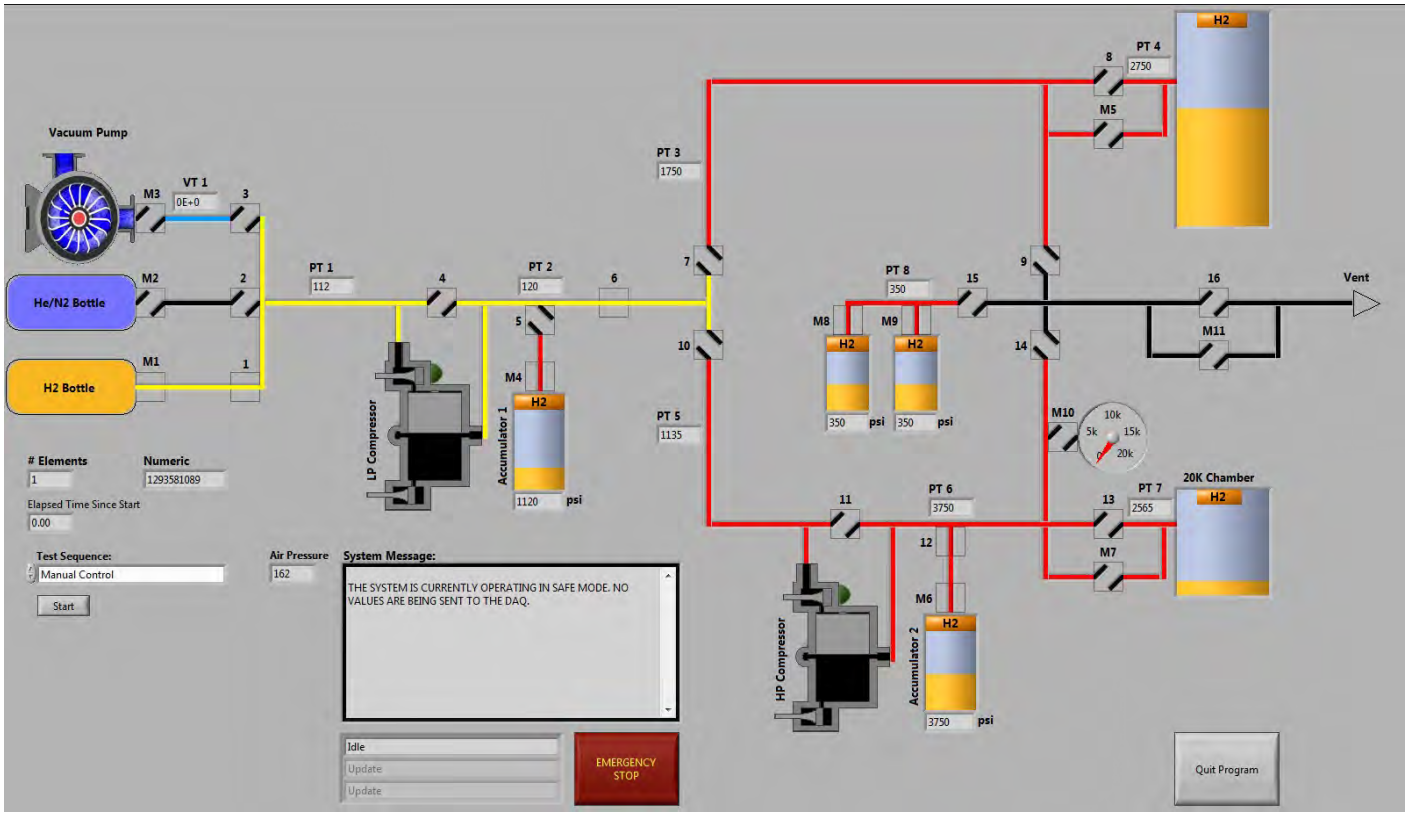

# Testing

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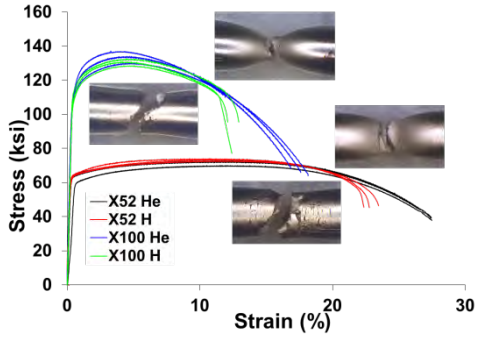
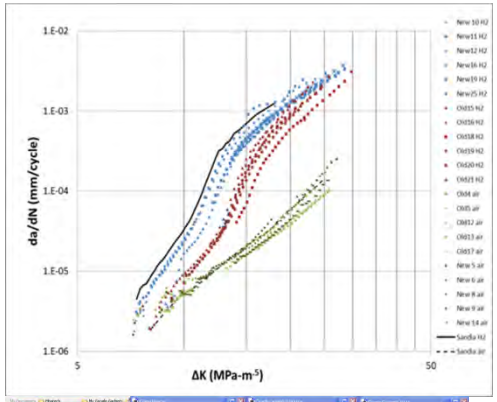
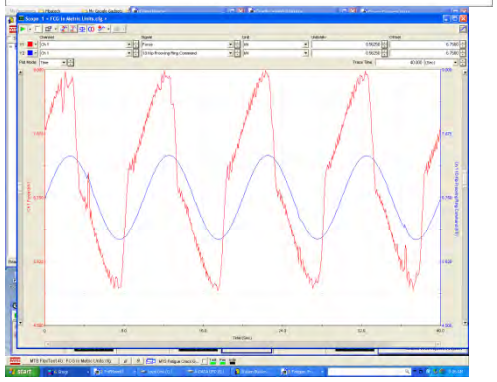
**Tensile: MTS Basic Testware**




**Fatigue: (10) MTS Multi-Purpose Testware**
**(1) MTS Multi-Purpose Testware**




**Why use an internal load cell?**



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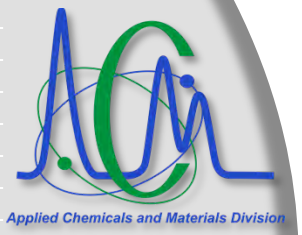
# Safety

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- Small Volume of Gas
- Large Building
- Ventilation
- Hydrogen Sensors
- “Fail-Safe” Design
- Minimize Spark Sources
- Explosion-Proof Camera
- Explosion-Proof Intercom between Hydrogen lab and control room
- ASME rated vessels
- Automatic, remote operation from control room, with manual overrides
- Hydrogen lab on far side of blast wall

# Safety

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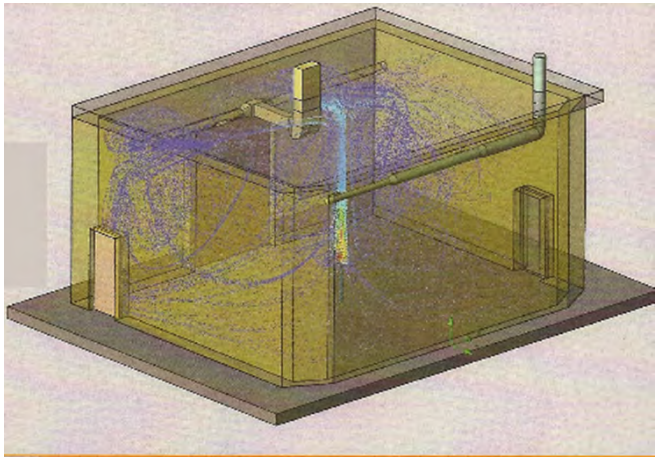
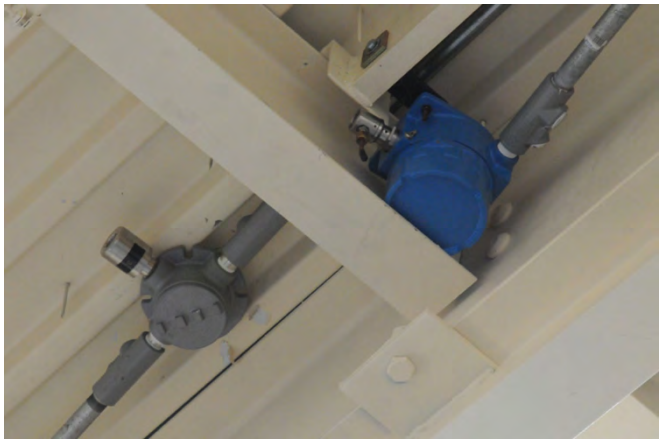
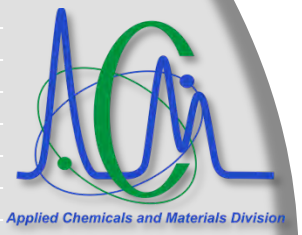
- Ventilation: 7 air changes per hour, 100 % outside air



- Hydrogen Sensors
  - Catalytic bead
  - Palladium thin-film



# Safety

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- "Fail-safe" design
  - Normally-closed air-operated valves
    - Backup manual valves
  - Explosion-proof or intrinsically-safe electronics in top 2/3 of lab height
  - System locks down upon ventilation loss, power loss, compressed air loss, or hydrogen alarm
  - Hydrogen-compatible materials

# Issues

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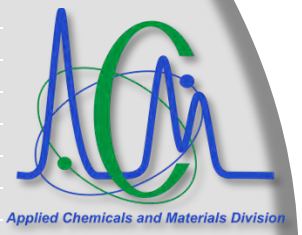
- Slow decompression
  - Sandia informed us of it
  - Initially used automatic valves and automated procedure to slowly decompress
    - Failures of automatic valves (stuck closed)
  - Currently using micro-metering valves and simpler automated procedure
  - Recalibrate clip gauges for each run
- High leak rate
    - Changed assembly procedure and increased torque on end-cap seal
- Pull Rods
    - Galling between pull rod and “slip washer”
    - Changed assembly procedure, see if it works

# Issues

- Strain-gauge-based load cell
  - <1% uncertainty
    - Mass of specimen string 180 to 220 N (40 to 50 lbs), friction and stiction of seals
    - Measure compliance for crack growth and load difference for stress intensity factor range
      - Both differential, relative measurements
- Gas Compressors
  - Require frequent diaphragm changes
  - Difficult to re-prime

# Issues

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- Remote Site
  - Power outages
    - At least 3 per year
    - Not enough power: add a load frame (pre-cracking)
  - Chiller
    - Too large, so it freezes
    - Use water instead
  - Air Compressor
    - Screw-type, high-reliability
    - Oil leaks, control switch failure

# Gas Contamination

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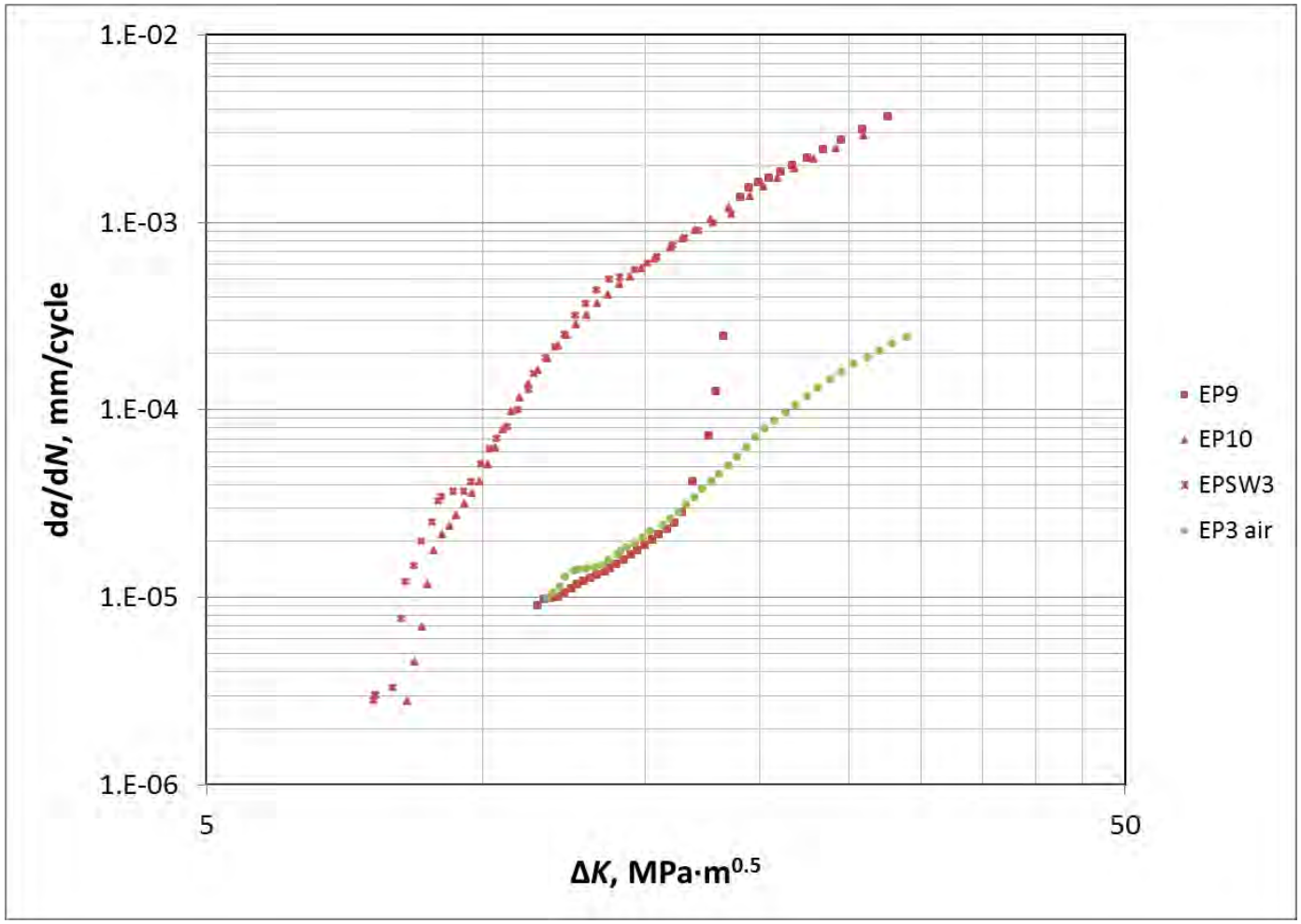

# Acknowledgements


- SNL Team: Brian Somerday, Chris San Marchi, Ken Lee, David Zanini, Kevin Nibur


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